

Dielectric Study of Cobalt Doped Potassium Hexatitanate ($K_2Ti_6O_{13}$) Fine-ceramics

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ABSTRACT

Pure and cobalt doped ($x=0.05, 0.10, 0.15$ mol %) polycrystalline potassium hexatitanate ($K_2Ti_6O_{13}$) ceramics were synthesized using conventional solid state reaction route. XRD result confirmed the successful doping of Co in the $K_2Ti_6O_{13}$ matrix. Further no other impurity peak was observed in the XRD pattern showing the single phase sample formation. Dielectric permittivity was found decreasing with frequency, it increased with increasing doping. While dielectric loss decreases with trivial doping, excessive doping caused their augmentation.

Keywords: Potassium hexatitanate; Solid State Reaction route; XRD; Dielectric Constant.

1. INTRODUCTION

A series of titanates having chemical formula $M_2O.n TiO_2$ (where $M = Li, Na, K$ etc. and $n = 2-8$) are well known. All of them have tunnel or layered crystal structures which are constructed of TiO_6 octahedra sharing edges with interlaying cations¹. Among the alkali titanates, potassium titanates ($K_2O.nTiO_2$) have several benefits compared with other ceramic whiskers. One of the most desirable

feature is the low cost of the whisker itself, which is one of the critical factor for commercial application. The potassium hexatitanate ($K_2Ti_6O_{13}$) with tunnel structure, relatively cheap fibrous material having various applications such as high thermal durability, chemical resistivity, dispersibility, in heavy metal ion removal, as ion exchangers and as photocatalytic materials²⁻⁶. In addition to other interesting technological applications such as ceramic capacitors, dielectric sensors, biosensors,

have been also reported for the $M_2Ti_nO_{2n+1}$ ($M = Na, K$ etc.)⁷⁻⁸. Properties of such materials may be modified by doping aliovalent 3d transition metal ions. Recently Cu doped $K_2Ti_6O_{13}$ is also reported by our research group⁹. In the present paper besides synthesis, dielectric properties of pure and Co doped $K_2Ti_6O_{13}$ ceramic has been studied.

2. EXPERIMENTAL

$K_2Ti_6O_{13}$ ceramic was prepared by conventional solid-state route taking stoichiometric amounts of the grinded AR grade K_2CO_3 and TiO_2 powders (Purity 99.9%), under acetone and calcined at $1000^\circ C$ for 24 h followed by furnace cooling. To prepare cobalt doped ($x = 0.05, 0.10, 0.15$) specimens, desired amount of cobalt oxide (Co_3O_4) powder (Purity 99.9%; AR grade) was added to the mixture of potassium carbonate and TiO_2 and the mass so obtained was recycled through the above process. The obtained powder was pressed

into pellets of 13 mm diameter and 1.00 mm thickness, which were further sintered at $1000^\circ C$ for 1 hour. X-ray Diffraction (XRD) spectrum on a X-ray powder diffractometer using Cu-K α radiations ($\lambda = 0.15406$ nm) in 2θ range from 10° to 70° with scan rate of $2^\circ/min$ is recorded. The dielectric measurements were carried out after applying the silver paste on the flat faces of the pellets, in the frequency range 100 KHz to 1000 KHz using LCR HI-Tester (HIOKI 3532-50). The loss tangent ($\tan\delta$) and parallel capacitance (C_p) of the pellet were measured directly from the impedance analyser. The value of dielectric permittivity (ϵ_r) is calculated using the formula:

$$\epsilon_r = \frac{C_p \times d}{\epsilon_0 \times A}$$

Where ϵ_0 is the permittivity of free space, d is thickness of pellet, A is the cross-sectional area of the flat surface of the pellet and C_p is the capacitance of the specimen in Farad (F).

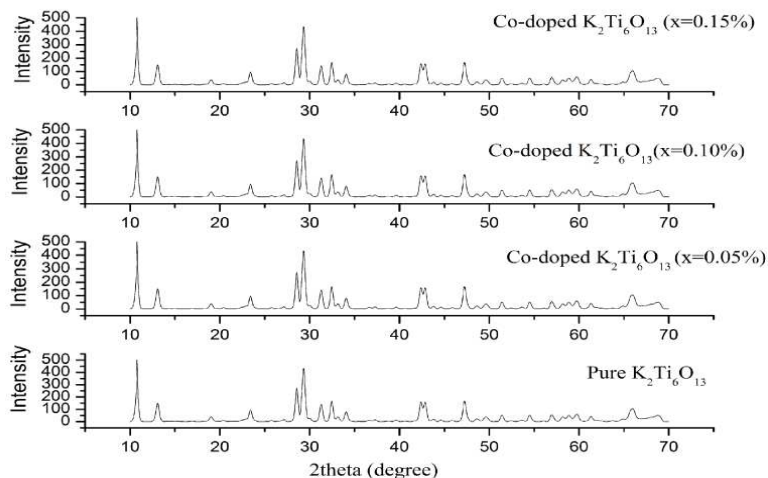


Figure 1: XRD pattern of pure and Co doped $K_2Ti_6O_{13}$

RESULTS AND DISCUSSION

Figure 1 shows the XRD patterns, achieved at RT, for pure and cobalt doped ($x = 0.0, 0.05, 0.10, 0.15$) $K_2Ti_6O_{13}$ ceramics. Further, no other impurity peak was observed in the XRD pattern showing the single phase sample formation, which was confirmed from ICDS card no.74-0275 and also confirms successful Co doping in $K_2Ti_6O_{13}$ matrix.

The dielectric constant is represented by $\varepsilon = \varepsilon_r - i\varepsilon''$ where ε_r is real part of dielectric constant (dielectric permittivity) and describes the stored energy while ε'' is imaginary part of dielectric constant, which describes the dissipated energy. Figure 2 (a) shows the frequency response of dielectric constant, recorded at RT, in the frequency range of 100–1000 kHz for $x = 0.0, 0.05, 0.10$ and 0.15 respectively, showing a consistent downfall in the permittivity with increasing frequency because at higher frequencies various polarisation processes ceases. The dielectric permittivity decreases for $x=0.05\%$, but it shows upsurge for $x=0.10$ & 0.15% . Increase in permittivity with rise in cobalt doping may be due to growth in the number of dipoles. Figure 2 (b) shows room temperature frequency response of $\tan\delta$ in the frequency range of 100–1000 kHz for all specimens. The loss tangent initially decreases for $x=0.05\%$, but further increases for $x=0.10$ due to inhibition of domain wall motion¹⁰, it augments for $x=0.15\%$ due to increase in leakage current. The increasing trend of $\tan\delta$ is due to space charges produced after all doping¹¹. Also $\tan\delta$ decrease with rise in frequency, proving that at higher frequency, ceramic specimen offer

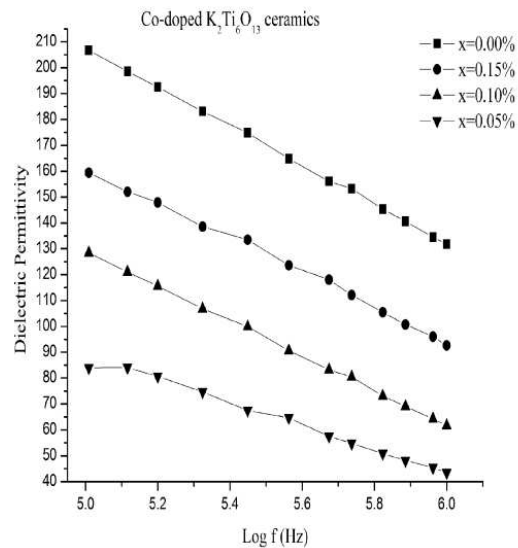


Figure 2 (a): Variation of dielectric permittivity with frequency at room temperature

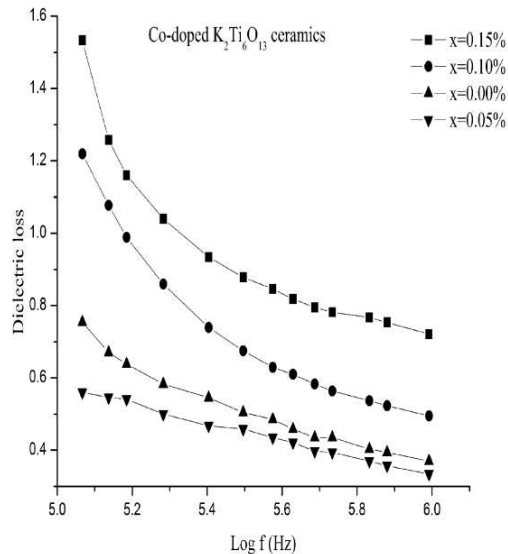


Figure 2 (b): Variation of dielectric loss with frequency at room temperature

low reactance to the ac signal and hence minimise the conduction losses¹². Therefore

amount of $\tan\delta$ decreases at higher frequency. These types of variations in the dielectric losses are the characteristic of the dipole mechanism and electric conduction¹²⁻¹³. Oxygen vacancies produced due to the substitution of Co ions at the host Ti sites, with charge compensating mechanism, cause deformation of the surrounding volume and modification of the local fields.

CONCLUSION

Cobalt doped $K_2Ti_6O_{13}$ ceramics were prepared by high temperature solid state reactions. Room temperature XRD pattern revealed the confirmation single phase formation of the sample. On cobalt doping primarily, losses decreased due to inhibition of domain wall motion and thereafter increased due to space charge polarisation. Dielectric permittivity decreases with frequency followed by increase for augmented doping, attributed to growing number of dipoles.

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